



**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

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TC 1700

In re application of:

DEBORAH FRANCES MIELEWSKI ET AL.

Group Art Unit: 1711

Examiner: Thao T. Tran

Serial No. 09/748,669

Filed: December 22, 2000

For: SYSTEM AND METHOD OF PREPARING A REINFORCED POLYMER  
BY SUPERCRITICAL FLUID TREATMENT

Attorney Docket No.: FMC 1289 PUS / 200-0661

**DECLARATION UNDER 37 C.F.R. § 1.131**

Commissioner for Patents  
U.S. Patent and Trademark Office  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:

I, Ellen C. Lee, do hereby declare and state as follows:

1. I, along with the other co-inventors, conceived of and reduced to practice the invention disclosed and claimed in U.S. patent application 09/748,669 in the United States prior to February 22, 2000.
2. As evidence of this conception and reduction to practice, attached is a redacted copy of an invention disclosure showing our conception and reduction to practice of the invention in the United States prior to February 22, 2000.
3. As further evidence of this conception and reduction to practice, attached is a redacted copy of an XRD log book. The last three entries on the first page of the log book and the entries on the second page of the log book are pertinent. Of

special note are the last two entries on the second page of the log book. These correspond to the Example in the '669 application.

4. Applicants therefore respectfully submit that the Japanese reference (JP 2000-053871 to Toru) is not a proper reference against the subject claims.

I hereby declare that all statements made herein are of my own knowledge and are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

8/15/2003

Date

Ellen Lee

Ellen Lee

AUG 23 2003

[http://www.fgti.ford.com/olid/cgi-bin/PrintAll\\_F\\_1.asp](http://www.fgti.ford.com/olid/cgi-bin/PrintAll_F_1.asp)

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Invention Disclosure Number: 17141  
Creation Date:  
Author: ELEE9

### Section 1: INVENTION DESCRIPTION

#### Title of Invention:

A MEANS OF PROCESSING A  
THERMOPLASTIC  
NANOCOMPOSITE MATERIAL  
WITH A SUPERCRITICAL FLUID  
IN ORDER TO ACHIEVE GOOD  
FILLER DISPERSION

#### Patent Evaluation Committee:

BODY

#### CPSC Code:

01.00.00

#### Originating Country Code:

US

#### Internal Reference Number:

None

#### Company:

FORD

### Section 2: PROBLEM & SOLUTION

Identify the purpose/function of the  
new technology(s) of the invention and  
advantages over prior technology:

Reinforcement of thermoplastics with  
fillers can result in materials with  
greatly increased mechanical properties.  
Thermoplastics are often too viscous to  
allow uniform dispersion of fillers.  
Furthermore, reinforcement by  
nanoscale clays requires good  
exfoliation of layered platelet structure.  
Our discovery allows good exfoliation,  
particle distribution, and dispersion of  
fillers within a thermoplastic matrix by  
processing with a supercritical fluid.

#### Attachments:

See Section:9 ATTACHMENTS

### Section 3: PRIOR ART

Identify the closest technology, if any,  
of which you are aware:

Toyota's in situ polymerization of nylon  
nanocomposites, Mucell foam  
technology, supercritical drying.

#### Attachments:

See Section:9 ATTACHMENTS

### Section 4: DETAILED DESCRIPTION

Please provide a full and complete  
description of the inventions:

Reinforcement of thermoplastics with  
fillers (glass, layered silicates, talc, etc.)  
can result in materials with greatly  
increased mechanical properties.

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Thermoplastics, however, are often too viscous to allow uniform dispersion and distribution of fillers. Furthermore, reinforcement by nanoscale clays (layered silicates, phyllosilicates, montmorillonite, etc.) requires good exfoliation or delamination of layered platelet structure. Our discovery allows good platelet exfoliation, particle distribution, and dispersion of fillers within a thermoplastic matrix by processing the mixture in situ with a supercritical fluid (SCF) such as supercritical CO<sub>2</sub>. Thermoplastic resin (polypropylene, nylon, etc.) is added to an extruder/injection molder with a desired amount of filler and CO<sub>2</sub> or other supercritical fluid. The polymer, swollen with SCF, undergoes a dramatic reduction in viscosity, which allows good filler dispersion. Layered silicate fillers can also be pretreated by SCF processing to obtain a card-house structure prior to addition. The filled thermoplastic (nanocomposite) may undergo slow or fast depressurization, and may result in a foamed material upon exiting extruder/injection molder.

See Section:9 ATTACHMENTS

**Attachment:**

**Section 5: DATES**

**First Recorded Description of Invention:**

E.C. Lee notebook — idea was recorded ) first XRD scan was run on samples :  
XRD on clay nanocomposite.  
(melt mixed with pretreated clay)

**Completed/Anticipated Completion Date of Working Model or Demonstrated Results:**  
**Planned/Anticipated First Production Use:**  
**Planned Usages:**

**Section 6: CATEGORY QUESTIONS**

**Invention Category:**  
**Category Questions do not exist or not answered:**

**Process:**

carbon	PDC ZR1 (4wt% P/DSC I) / 1150C / redox / 100C normal slits: 40ms / 45kV powder / ethanol on new $\text{SrTiO}_3$	x2GWG651
"	( " ) + 90° rotation	x2GWG652
"	( " ) + remove 0.5mm skin	x2GWG653
A. Drews	radiator fin piece from K-Lazurcz, self supported 13°-65° / 0.25° / 2 sec normal slits 45kV 40ms	hdx2.004
S. Fenner	$(\text{CeZr})\text{O}_2$ L-990828 For Y. Cheng slits: 2.38 → 1-0.3 Cvt 40ms / 45kV cont scan 1°/in 5°-90° 0.03 chopper	safX2001
S. Fenner	$(\text{ZrCe})\text{O}_2$ ZC80201 - m <sub>ZrO2</sub> / m <sub>CeO2</sub> = 80/20 For Y. Cheng. Same conditions as safX2001	safX2002
EClee	untreated clay (INSU) I-30E Nanocor slits: 0.48 → 0.2, 0.1 1.5°-15°; 0.03°; step; 3 sec cl time 45kV 40ms (vaseline on ZBO)	ECLX2024
EClee	same as above; pre cl. time 4s (vaseline on ZBO)	ECLX2025
EClee	super critical $\text{CO}_2$ treated I-30E (1700psi, 80°C, 5h) same conditions as ECLX2024. (vaseline on ZBO)	ECLX2026

USER	DESCRIPTION	DATE	FILENAME
EClee	Supercritical CO <sub>2</sub> / PDMS ( <del>20wt%</del> ) treated clay 20wt% PDMS / 80% clay, excess PDMS vented w/ CO <sub>2</sub> same cond. as ECLX2024 (Vaseline on ZBQ)		ECLX2027
EClee	untreated I-30E on ZBQ (no Vaseline) same cond. as ECLX2024		ECLX2028
EClee	supercritical CO <sub>2</sub> / PDMS treated clay (same sample as ECLX2027) on ZBQ same conditions as ECLX2024	"	ECLX2029
EClee	supercritical CO <sub>2</sub> treated I-30E (same sample as ECLX2027) on ZBQ Same cond. as ECLX2024	"	ECLX2030
A. Owens	ultrasonically removed fluoride crystals from a jet radiation film, then filtered (.45mm) 5°-90° / .025" / 2sec 45kV 40mA suspension deposited on ZBQ		ARDX2.005
EClee	5% scCO <sub>2</sub> / PDMS clay - PB523 microtomed on ZBQ 1.5°-15°: 0.033" step: 3 sec normal <del>slits</del> 0.48 → 0.2, 0.1 45kV 40mA		ECLX2031
EClee	5% scCO <sub>2</sub> clay + 10% PB + GS23 microtomed on ZBQ same as above		ECLX2032